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International Standardization in Particle Characterization for Quality and Safety Assessment in Particle Technology

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Abstract

The interdisciplinary field of particle characterization and particle release characterization, especially regarding environmental, health and safety aspects associate with nanotechnology, requires coordinated actions in standardization between measurement methods and application scenarios. This in turn, demands also a consistent terminology in context with nanotechnology. Established liaisons, represented by experts, between international standardization committees try to ensure detailed coordination in the daily work.

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Keywords: *Standards; ISO; particle characterization; nanotechnology; nanoparticle release*

1. Introduction

According to EN 45020:2006 [1], standardization is the "activity of establishing […] provisions for common and repeated use aimed at the achievement of the optimum degree of order in a given context" by "formulation, issuing and implementing standards" that were "acknowledged by a majority of representative experts" of industry, research and regulatory bodies. Therefore, the aim of standardization is "to make a product, process or service fit for its pur-

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pose". In this context, the focus can be based on one or more goals, e.g. "variety control, usability, compatibility, interchangeability, health, safety, protection of the environment, product protection, mutual understanding, economic performance, trade". Standards are voluntary, i.e. there is no automatic legal obligation to apply them. However, laws and regulations may refer to standards and even make compliance with them compulsory.

The World Standards Cooperation (WSC) can be seen as the umbrella organization in standardization. The WSC is the union of the three international standards organizations, i.e. the International Organization for Standardization (ISO) with currently 162 member states worldwide, the International Electrotechnical Commission (IEC) and the International Telecommunication Union (ITU). Beside the international standards organizations there are regional standards organizations. For Europe, these are the European Committee for Standardization (CEN), the European Committee for Electrotechnical Standardization (CENELEC) or the European Telecommunications Standards Institute (ETSI). International standardization organizations are networks of national standard bodies, like the Association Française de Normalisation (AFNOR) or the German Institute for Standardization (DIN). The European national member bodies have to adopt CEN-standards to national standards, whereas they can decide to adopt ISO-standards.

The particle technology covers a wide range of industrial application in production and processing. The improvement or development of particle production processes and customized products properties taking also into account aspects regarding environment, health and safety (EHS) requires a profound understanding of particulate interactions. This in turn demands also technologies for particle system characterization and a unified terminology for a worldwide communication. Accordingly, particle technology is established in national and international standardization bodies since theirs founding.

Nanotechnology is the "application of scientific knowledge to manipulate and control matter in the nanoscale […] in order to make use of size- and structure-dependent properties and phenomena, as distinct from those associated with individual atoms or molecules or with bulk materials" [2]. It is evident, that the nanotechnology represents a more and more important part of particle technology. Due to the wide field of application in product-improvement and product-development, the nanotechnology is an increasingly growing industrial sector

Table 1. Technical committees (TC) and subcommittees (SC) of ISO, IEC and CEN as potentially involved for standardization activities regarding nanotechnologies and nanomaterials.

organization	TC / SC	main responsibilities
ISO	TC 24 / SC 4	Particle characterization
	TC 142	Cleaning equipment for air and other gases
	TC 146 / SC 2	Air Quality – Workplace Atmospheres
	TC 194	Biological evaluation of medical devices
	TC 201	Surface chemical analysis
	TC 202	Microbeam analysis
	TC 229	Nanotechnologies
	TC 256	Pigments, dyestuffs and extenders
IEC	TC 113	Nanotechnology standardization for electrical and electronic products and systems
CEN	TC 137	Assessment of workplace exposure to chemical and biological agents
	TC 138	Non-destructive testing
	TC 162	Protective clothing including hand and arm protection and lifejackets
	TC 195	Air filters for general air cleaning
	TC 230	Water analysis
	TC 248	Textiles and textile products
	TC 352	Nanotechnologies

. The nanotechnology has therefore found its way into national, regional and international standardization as exemplarily shown in Tab.1 by identified standardization activities regarding nanotechnologies and nanomaterials in ISO, IEC and CEN.

In 2010, the European Commission (EC) mandated the European Committees for Standardization (i.e. CEN, CENELEC, ETSI) for standardization activities regarding nanotechnologies and nanomaterials (M/461 EN) [3]. For the purpose of coordination between the different international technical committees (TC) within and between ISO and CEN, ISO/TC 229 “Nanotechnologies” founded the Nanotechnologies Liaison Coordination Group (NLCG). Within the CEN, the CEN/TC 352 “Nanotechnologies” took the leadership for the coordination in the execution on the EC-Mandate.

Based on the identified standardization activities, three important key issues in international standardization can be derived, i.e. terminology, nanoparticle characterization and nanoparticle release characterization. The mentioned key issues will be briefly presented and discussed in the following.

2. Fundamentals

2.1. Representation and definitions regarding the particle characterization of particulate systems

The particle technology respectively the nanotechnology is an interdisciplinary field and extends over a variety of different disciplines in science and industry. Therefore, numerous scientific studies suffer from an inappropriate representation of particle measurement results that lead sometimes to misinterpretation of measurement data. Within the ISO, standardization activities regarding particle characterization are performed by the technical committee ISO/TC 24 that consists of two subcommittees (SC), i.e. SC 4 “Particle Characterization” and SC 8 “Test sieves, sieving and industrial screens”. The nanoparticle characterization is covered by several working groups (WG) within ISO/TC 24/SC 4 in liaison to ISO/TC 229 and CEN/TC 352.

The basis of granulometric analyses is a consistent representation of particle measurement results. For this purpose, ISO 9276 was already established in 1990 and since this time continuously maintained by the ISO/TC 24/SC 4/WG 1. ISO 9276 [4] consists of six parts and addresses therein the graphical representation of size distributions, the calculation of mean diameters, the fit to distribution models, the characterization of classification processes, the properties of logarithmic normal distributions and a collection of macro- and mesoshape descriptors.

A vocabulary on particle characterization with more than 250 definitions from A like adsorbate to Z like zeta-potential, which are included within more than 30 published ISO standards, was recently released as ISO 26824:2013 [5]. The corresponding definitions are freely provided by the ISO at the ISO Online Browsing Platform (https://www.iso.org/obp) [6].

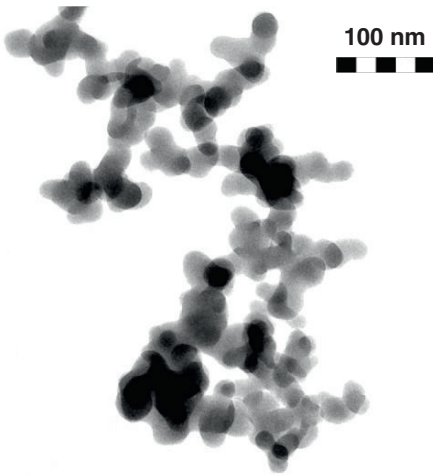
2.2. Terminology on nanomaterials and nano-objects

The ISO/TC 229 "Nanotechnologies" has defined a science-based terminology for nanoscale, nanomaterial, nanoparticle and nanostructured material in ISO/TS 80004-1:2010 [2] and ISO/TS 27687:2008 [7]. ISO/TS 27687:2008 [7] is currently under revision and will be allocated as ISO/TS 80004-2. These ISO standards differentiate between nano-objects and nanostructured materials. The identifying feature of nanostructured materials is that their internal or surface structure is in the nanoscale (≤ 100 nm), but their external dimensions are typically greater. Nano-objects (i.e. nanoparticles, nanoplates and nanofibres) have three, two or one external dimension in the nanoscale. The definitions were developed in cooperation with the Working Party of Manufactured Nanomaterials (WPMN) of the Organisation for Economic Co-operation and Development (OECD).

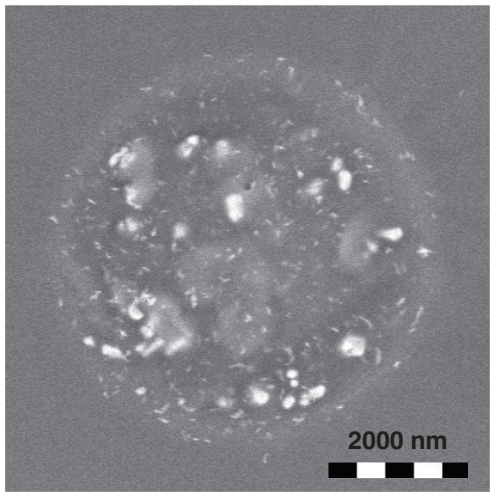
Currently, no harmonized definition of the term nanomaterial exists under regulatory aspects, which is necessary to address the question, how much nano-object content according to the ISO definition makes a normal material to be classified as a nanomaterial. In October 2011, the EC has issued a recommendation on the definition draft of the term nanomaterial (2011/696/EU) [8]. This definition comprises "natural, incidental or manufactured materials containing particles in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimension is in the size range 1 nm - 100 nm". Paragraph 12 of the "Whereas" section of the recommendation "also includes particles in agglomerates or aggregates whenever the constituent particles are in the size range 1 nm – 100 nm". Further notes on the EC-recommendation on the term nanomaterial are given in [9]. It should be kept in mind, that the definition draft is based solely on the size of the constituent particles of the material, without regard to outside aggregate sizes, which are relevant for exposure and hazard properties.

3. Standardization in (nano)-particle characterization

Fig. 1 shows for example two different kinds of complex-nanostructured particles, which fulfill the EC recommendation on the definition of nanomaterials. It is evident that it will be difficult to characterize such particle systems by conventional particle measurement technologies other than imaging methods like scanning electron microscopy (SEM) or transmission electron microscopy (TEM).



a) TEM-image of a synthetic and fractal SiO_2-aggregat (≈ 500 nm) containing of sintered nanoscaled primary particles (≈ 18 nm)



b) SEM-image of a dried spray droplet (≈ 5µm) made of acrylate topcoat with embedded TiO_2 pigment particles (≈ 200 nm) and embedded iron oxide nanoparticles (< 100 nm)

Fig. 1. Examples of complex-nanostructured particles.

The extent to which the EC-recommendation on the nanomaterial definition can be realized by available measurement technologies is the aim of the currently started European research project "NanoDefine" [10]. In fact, there are many scientific and technical problems [e.g. 11], which should be solved before regulatory activities.

As mentioned above, standardization in nanoparticle characterization is performed within ISO/TC 24/SC 4. Additionally to imaging methods for morphology inspection of single particles, aerosol measurement devices have some benefits for exposure analysis compared with particle measurement techniques for liquid dispersions (i.e. emulsions, suspensions or combinations of them), for instance the ability of providing absolute count numbers or the independency from specific material properties (e.g. from the index of refraction). A fundamental aerosol measurement principle that allows the characterization of particles down to a view nanometre is the electrical mobility analysis as

described within ISO 15900:2009 [12]. One problem from metrological view, which still exists for aerosol measurement technology, is the lack of a concentration reference material. An important step in this direction represents the international standard draft (DIS) ISO/DIS 27891:2013 [13], where the calibration of condensation counters is focused.

In the field of granulometric techniques for liquid dispersion characterization, a fundamental challenge is the characterization of the dispersion stability, i.e. "the absence of change in specified properties over a given timescale" [14]. Therefore, the technical report ISO/TR 13097:2013 [14] was issued by ISO/TC 24/SC 4/WG 16 and gives (in the absence of a direct measurement) two different approaches to determine relative property changes. Especially in larger cluster research projects, dealing with fate, exposure and hazard of nanomaterials the sample preparation turned out to be the deciding step, e.g. for risk assessment of TiO_2 [e.g. 15]. Zeta potential measurement proved to be a necessary tool for checking dilution and stabilization protocols. Therefore, ISO/TC 24/SC 4/WG 17 issued methods for zeta potential determination within ISO 13099 [16], which consists currently of two standards and one final draft of an ISO standard (FDIS). Regarding the addressed preparation preconditions comparable and reproducible particle/agglomerate size measurement by hydrodynamic mobility analysis (e.g. by dynamic light scattering - DLS) can be achieved [e.g. 17].

The European coordination platform "NANOfutures" and also short term projects like "Value4Nano" or "nanoSTAIR" try to focus the research to industrial application problems in nanotechnology by managing the link to standardization and regulation issues, e.g. for risk management an life cycle analysis. One important issue is the exposure.

4. Standardization in (nano)-particle release characterization

Based on their specific properties and high mobility, the use of nanomaterials is associated with possible risks for environment, health and safety [e.g. 18]. Risks on EHS depend on both material toxicity and material exposure [e.g. 19]. Considering health effects, the nanoparticle uptake by inhalation is seen as the most critical one. Systematic studies on nanomaterials in laboratory can provide basic information about the ability and the quantity of nano-object release into the air for exposure estimation [18]. Comparing potential release scenarios for airborne particulate emissions [e.g. 20, 21] with performed studies on airborne nano-object release characterization [e.g. 22-31] shows that the whole life cycle of nanomaterials, which comprises also the different types of dispersion (i.e. nanostructured powders, fluid nano-dispersions and nano-composites), is covered in most instances. The performed studies on airborne particulate release have shown a strong dependence on the sample material, the sample composition and the sample condition and especially on the applied processes.

Kept in perspective, published studies regarding nano-object release into air suffer more or less from three key issues, i.e. a consistent terminology, standardized metrological procedures and the kind of data evaluation. Thus, a quantitative comparison between the different studies is often difficult or impossible, if necessary parameters are missing. This in turn, affects furthermore a consistent trace back of measurement data to real exposure situations. To fill this gap, ISO/TC 229/WG 2/PG 10 has developed in a first step the technical specification (TS) ISO/TS 12025:2012 [32], which is a general framework for determining airborne release of nano-objects from nanostructured powders by means of aerosol analysis. The TS provides information on the methodology for nano-object release quantification that covers beside necessary measurands and process parameters also the presentation of measurement results by specific release numbers. ISO/TS 12025:2012 [32] supports also standardization regarding nano-object release testing based on nanocomposites, e.g. abrasion procedures lead to powdered wear prior a possible aerosolization.

The scientific findings from nano-object release studies performed on nano-object-doped or pigmented coatings and plastics [e.g. 25-31] currently integrated by ISO/TC 256/WG 2 "Nanotechnological properties of pigments and

extenders" within the working draft (WD) ISO/WD 00004:2013 [33]. In detail, the WD focuses on defined scenarios for release testing with regard to sensitivity and reproducibility of standardized particle measurement methods.

5. Conclusion

The current nanotechnology key issues of both ISO and CEN are the standards development for a uniform terminology and for relevant measurement methods for material and process characterization by coordination between the different technical committees of international and national organizations. For this purpose, the Nanotechnologies Liaison Coordination Group (NLCG) was established by the ISO/TC 229.

Beside terminology and measurement methods, a further important point in current nanotechnology standardization is the risk management regarding environment, health and safety. Standardization in exposure and hazard quantification becomes increasingly important. This in turn leads to the central question of the nanomaterial definition for regulatory purposes and their relation to toxicological and exposure studies. On the European level, the latter one is also requested in the substance accreditation process (REACH).

Apart from granulometric analyses by imaging methods (SEM, TEM), the metrological determination of characteristic properties allowing the classification of a material as a nanomaterial in accordance with the recommendation of the European Commission is still a complex scientific and technical challenge. From our perspective, the measurement of aggregate/agglomerate size distributions under defined conditions (e.g. dispersing procedures, release scenarios) seems to be still essential for characterizing particulate systems.

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